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*An account of a new Fulminating Silver, and its application as a Test for Chlorine, &c.* By EDMUND DAVY, F.R.S., M.R.I.A., &c., *Professor of Chemistry to the Royal Dublin Society.*

Read May 23, 1831.

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I LATELY had the honor of sending to the Royal Irish Academy, a paper of mine, "On a new Acid and its Combinations," published by the Royal Dublin Society. Whilst it was printing, I found that some of the compounds therein described, spontaneously exploded when brought in contact with chlorine gas. Such unexpected results appeared sufficiently interesting to merit further inquiry: accordingly, I instituted a series of experiments on the subject; and as they have led to a number of new facts which may admit of useful applications, I venture to submit some of them to the consideration of the Academy. In the present communication, I purpose to give a brief account of a new fulminating silver I have obtained, and of the principal experiments which illustrate its efficacy as a test for chlorine.

1. *Modes of making the new Fulminating Silver; its properties and composition.*

This compound may be readily prepared from Howard's well-known fulminating mercury by the following simple processes:—Put any quantity (suppose from ten to fifty grains or more,) of Howard's compound, either in a dry or moist state, into a phial, with about half an ounce of pure water, and about twenty grains of zinc filings, or granulated zinc, for every ten grains of Howard's compound used. Cork the phial and occasionally agitate its contents, for about twenty or thirty minutes. Less time will answer if the water is moderately warm. The fluid is fulminate of zinc; filter and treat it with nitrate of silver, the white precipitate which occurs is the new fulminating silver. This substance may also be easily made, by adding nitrate of silver to an aqueous solution of any of the soluble fulminates I have described in the paper already alluded to, or from Howard's fulminating silver by the means recommended above, in the case of the fulminating mercury; and after being collected on

a filter, washed and dried, either in the open air or at a heat not exceeding  $212^{\circ}$  Faht. its properties appear to be the same.

The new fulminating silver, in drying on the filter, breaks into small lumps, which are easily separated from the paper, and reduced by the gentle pressure of a platina spatula, or of a card, to an impalpable powder. Its colour, when recently prepared, is white; and if dried in the dark, or in a weak light, it retains this colour for some time. On being exposed to a strong light or the sun's rays in a moist state, it soon undergoes progressive changes of colour; from greyish white to yellowish brown, brick red, blackish brown, and even black. These changes are facilitated by the presence of water, and they appear to be connected with the partial decomposition of the compound; for on exposing a little of it in water for some time to the action of the solar rays, I observed, by means of a magnifying glass, minute specks of metallic silver on the surface of the water, apparently carried there by small globules of gas, which were occasionally evolved from the compound.

The new fulminating silver, when heated to about  $350^{\circ}$  Faht. assumes a darkish tint and explodes, producing a large yellowish flame, and a loud report. It also explodes by percussion, when struck with a hammer on an anvil; and by friction, when it is rubbed between two hard surfaces; hence, it must be handled with caution.

It is insoluble, or very nearly so, in cold water. I tried in vain to dissolve a single grain of the dry compound in about eleven fluid ounces of pure water; after some days, the greater part was undissolved. The water, however, became slightly turbid, acquired a metallic taste, and on evaporating some of it to dryness, a very minute portion of the compound remained. Boiling water dissolves a certain limited quantity of the compound; but as the fluid cools, the greater part separates in minute crystals, which are principally long needles, intersecting each other.

The new fulminating silver, in a dry state and even whilst it yet retains moisture, instantly explodes when brought in contact with pure chlorine gas; and also when this gas is mixed with most other gases, as will be presently stated.

Muriatic acid decomposes the new fulminating silver, readily, converting it into chloride of silver, whilst hydrocyanic acid is evolved; and if the experiment is continued to dryness, sal-ammoniac rises in vapour, and the pure chloride of silver remains.

Strong nitric acid gradually decomposes the new fulminating silver with evolution of gas; but if diluted, fulminic acid is slowly disengaged, and nitrate of silver formed.

Strong sulphuric acid readily explodes the new compound; but when diluted, the fulminic acid is gradually evolved, and sulphate of silver produced.

The new compound is soluble, to a certain extent, in liquid ammonia, and as the alkali evaporates, minute crystals are deposited, which explode by heat, but not in chlorine gas. Solutions of the fixed alkalies dissolve a portion of the new compound, and form crystallized fulminating compounds, which I have not examined.

The new fulminating silver readily explodes by the electric spark, producing a loud report, and a reddish flame. I repeatedly succeeded in the experiment, by simply placing a little of it on the prime conductor, bringing a brass ball near it, and putting the machine in motion. I found that the very feeble charge remaining on the prime conductor, after giving the machine a few turns, was quite sufficient to explode it.

The new fulminating silver is decomposed by a number of metals, as zinc, iron, copper, &c. in cases when it is put into water, and these metals severally introduced; new fulminating compounds of each metal are in a little time produced.

As the new fulminating silver may be formed from Howard's fulminating silver, and may be readily converted into chloride of silver, (as has been stated;) little difficulty was anticipated in ascertaining its composition; and yet from the precautions to be observed, I made several experiments, (using both methods,) before I obtained any satisfactory results. In converting Howard's fulminating silver into the new compound, a given weight of the former, well dried, was very cautiously put into a small phial, (nearly filled with pure water,) with about twice its bulk of fine zinc filings. The contents of the phial were occasionally agitated gently, and after some hours the fluid, (fulminating zinc,) was filtered, and carefully treated with solution of crystallized nitrate of silver, until no farther precipitate took place. The new compound thus produced was then thrown on a filter, washed, dried at about 212° Faht. and weighed. In one experiment thus conducted, 1.41 grain of Howard's fulminating silver, afforded 0.87 grain of the new fulminating silver; but a portion could not be separated from the filter, which being dried, broken in pieces and heated, afforded successive explosions. The loss thus sustained, may be estimated at 0.03 grain, which being added to the 0.87 make 0.90. Making these results the basis of calculation, 100 grains of Howard's compound would afford about 63.8 grains of the new compound; for  $1.41 : 0.90 :: 100 : 63.8$  nearly. Now, according to my analysis, 100 grains of Howard's compound, contain 26.25 grains of fulminic acid, and  $63.8 : 26.25 :: 100 : 41.14$  nearly. Hence, 100 grains of the new compound would consist of

$$\begin{array}{r} 58.86 \text{ oxide of silver} \\ 41.14 \text{ fulminic acid} \\ \hline 100.00 \end{array}$$

and taking from my experiments the proportional number of fulminic acid as 42. hydrogen being unity: the new compound would consist of two proportions of fulminic acid  $2 \times 42 = 84$  and one proportion of oxide of silver 118, for

$$58.86 : 41.14 :: 118 : 83.7$$

In a second experiment, conducted like the first, 5.45 grains of Howard's compound,

\* On a new acid and its combinations. *Trans. Royal Dublin Society*, 1829.

were converted into the new compound ; but only 3.02 grains of this substance could be collected from the filter, instead of about 3.48 grains : the loss on the filter, however, was nearly equivalent to the deficiency, as was ascertained by comparing the weight of the dried filter, with another, equal in every respect, and placed under similar circumstances.\*

The preceding experiments afford the nearest approximations I could obtain, to the composition of the new compound, by that mode of operating. The difficulty of gaining uniform results, by converting Howard's compound into the new compound, arises partly from the facility with which the new compound, when first formed, is redissolved by an excess of nitrate of silver ; and partly from its being to a certain limited extent, either dissolved by water, or partially decomposed by it.

In converting the new compound into chloride of silver, a given weight of it, (well dried,) was put into a platina crucible ; a little pure water was added, and then some pure muriatic acid of moderate strength ; a considerable action took place, hydrocyanic acid appeared to be evolved, and after digestion for a short time, water was put into a crucible, the chloride was thrown on a filter, washed, dried, collected and fused in a platina capsule, previously counterpoised in a very delicate balance. Operating in this manner, I found, that on fusing the chloride in the capsule it was of a dark colour, having some small specks in it like charcoal. On exposing the capsule to a full red heat, the dark coloured chloride seemed to undergo ebullition ; it evolved gas, and gradually assumed a yellowish white colour. It now lost no weight on being heated to redness, and no part of it appeared to be reduced. In one experiment, thus conducted, 6 grains of the new compound afforded 4.70 grains, and in another experiment, 6 grains yielded 4.82 grains of fused chloride of silver. Now, if the mean of these experiments is taken, 6 grains will afford 4.76 grains, and 100 grains of the new compound, will yield 79.33 grains of chloride, equivalent to 54.73 grains of metallic silver, or 58.44 grains of oxide of silver. These results, so nearly correspond with those derived from a different method of examination already noticed, that I venture to regard the new fulminating silver, as a compound of one proportion of oxide of silver 118. and two proportions of fulminic acid 84. or of

58.42 oxide of silver

41.58 fulminic acid

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100.00

I made a number of comparative experiments on the two fulminating silvers, all of

\* I have since found, that in decomposing Howard's compound, by Zinc, &c. another compound of silver is formed. It is of a dull white colour, but changes on exposure to light. It does not explode by heat, but ignites and burns for an instant, leaving a brown substance, which by a strong heat is converted into silver. When decomposed over mercury, the products were oxide of silver, carbonic acid gas, and carbonate of ammonia. This substance may partly account for the loss in the above experiments ; but its quantity seems too minute to affect the accuracy of the statements made.

which tended to prove that they are different compounds. Thus, I found that the new compound, exploded when heated from about  $350^{\circ}$  to  $370^{\circ}$  of Faht.; whilst Howard's compound required an increase of  $100^{\circ}$  or from about  $450^{\circ}$  to  $470^{\circ}$  Faht. to explode it. Howard's compound readily dissolves in boiling water, as M. Liebig observed, but the white silky crystals are rapidly deposited as the solution cools, and very little remains in solution at the temperature of the air. The new compound is nearly insoluble in boiling water; I could not dissolve half a grain of it in several ounces of water, kept boiling for some time in a platina crucible. Even the small portion that dissolves is found to have acquired new properties, for on being collected and dried, it will not explode in chlorine, and is probably by a loss of acid, converted into Howard's compound.

Howard's compound, however carefully prepared and dried; whether in crystals, or an impalpable powder, does not spontaneously explode in chlorine gas, or in mixtures of this gas with other gases. Whereas, the new compound, whether dried at  $60^{\circ}$  or at  $212^{\circ}$  and even before it becomes pulverulent from loss of moisture, readily explodes under such circumstances.

According to my experiments, Howard's compound contains one proportion of fulminic acid, and one proportion of oxide of silver; and it has been called fulminate of silver: as the new compound appears to contain two proportions of the acid, and one of the oxide, it is properly a bi-fulminate of silver.

##### *5. Experiments on the application of the new Fulminating Silver, as a test for Chlorine, &c.*

The chlorine gas employed in the following experiments, was generally made in the usual way, by adding diluted sulphuric acid to a mixture of common salt, and black oxide of manganese. Occasionally, it was procured from muriatic acid and the same oxide. Sometimes the gas was received over water, at other times it was collected in dry bottles furnished with ground stoppers. The new fulminating silver, or test, as I shall now, for convenience, call it, was made by decomposing fulminating zinc by nitrate of silver, as has been stated. It was sometimes dried on a sand bath at a temperature not exceeding  $212^{\circ}$  Faht., and sometimes in the open air. It was always, however, sufficiently dry to be pulverulent. It was commonly used in very minute quantities; a simple grain serving, on an average, for upwards of fifty, and occasionally for about one hundred separate experiments. It indicated the presence of chlorine, by instantly exploding when brought in contact with this gas, and also with other gaseous mixtures in general, which contained chlorine.

1. The test readily and repeatedly exploded when put into bottles of very impure chlorine gas, made a long time and having no vestige of colour; and also into bottles

of the gas containing about one-tenth their bulk of water, which had been exposed last summer, during several weeks, in the open air, to light and the sun's rays.

2. Water recently saturated with chlorine in one bottle, was partly transferred to another bottle. On agitating the fluids in both bottles, and introducing the test, it instantly exploded. Similar experiments were made with a saturated solution of the gas which had been excluded from light for a month, and the results were precisely similar.

3. Mixtures, both of pure and impure chlorine and hydrogen, being treated with the test, instant explosions took place, and the production of muriatic acid gas.

4. On adding the tenth of a cubic inch of nitric acid to an equal bulk of muriatic acid, chlorine gas was presently evolved; and the test, on being applied, repeatedly exploded. In cases where those acids were strong, and had acted for a short time on each other, it was only necessary to put the test on a slip of platina, and bring it to the mouth of the glass containing the acids, when an explosion took place, and the effect was produced several times. I found, indeed, that two drops of strong nitric, and one of muriatic acid, put into the same glass, produced a sufficient quantity of chlorine to explode the test several times.

5. Nitric acid being added to common salt, both in its usual state of dryness and also after being fused, the gas evolved readily, and repeatedly exploded the test. The results were precisely similar, when nitric acid was added to a number of dry chlorides, as those of potassium, lime, iron, &c. and the test applied.

6. Strong sulphuric acid was added to chloride of lime made above twelve months since; chlorine gas appeared to be evolved and the test exploded. Fluid chloride of magnesia made some time before, being treated with sulphuric acid, gas was disengaged which exploded the test.

7. A number of experiments were made to ascertain whether the power of the test would be injured or destroyed by diluting chlorine with other gases. Thus nitrogen, nitrous oxide, nitrous gas and carbonic oxide gases were mixed in different proportions with chlorine, but in every instance the test exploded with great facility. When olefiant and carburetted hydrogen gases, were separately mixed with certain proportions of chlorine, the test simply exploded when brought in contact with the mixtures; but when the same gases were severally mixed with chlorine in the proportions of about equal volumes of each, and the test applied, the mixed gases inflamed the instant the test exploded, and the interior of the tube was completely blackened from the deposition of carbon.

8. Different acid gases, as muriatic acid gas, nitrous acid gas, and carbonic acid gas, were separately mixed with chlorine gas, without any regard to proportions; but this circumstance neither prevented, or retarded in any degree, the action of the test, which instantly and repeatedly exploded on being dropped into the respective mixtures. Even gases which are known to be rapidly acted on by chlorine, as sulphu-

retted by hydrogen and sulphureous acid gas, did not hinder the action of the test provided there was the slightest excess of chlorine present.

9. Melted sulphur, and phosphorus were put into bottles of chlorine, and after the respective chlorides were formed, the test being repeatedly introduced into the bottles, in every instance, it exploded.

10. The protoxide of chlorine was collected in an open tube, and a bit of the test introduced; a double explosion took place; first of the test and then of the gas. The test being now added, it exploded as in a mixture of chlorine and oxygen. The peroxide of chlorine being treated with the test, a much louder explosion took place than in the foregoing experiment.

11. A number of experiments were made to ascertain whether the vapours of different fluids diffused through chlorine gas would prevent the usual action of the test. The chlorine gas was agitated in contact with water heated nearly to the boiling point, and whilst the hot vapour issued from the bottle, the test was several times applied, and in every trial it instantly exploded.

12. Sulphuric ether was put into a bottle of chlorine gas, and agitated; a bit of the test being now added, it exploded; inflammation took place, and carbonaceous matter was deposited on the sides of the bottle. A similar experiment being made with alcohol, the test exploded several times.

13. A few drops of oil of turpentine and of naphtha were separately put into phials of chlorine gas, a rapid action of course took place, and much heat was produced. The test, on being instantly applied, exploded in both phials; but if a short interval was suffered to elapse, and the fluids were agitated, the chlorine was all absorbed and no effect was produced on the test.

14. Strong muriatic, nitric, acetic, and hydrocyanic acids, were separately put into phials of chlorine gas and agitated; the test being repeatedly applied to all the phials, exploded in every instance. The test did not explode in any of those acids, or their respective vapours: nor in aqua regia, but when the deep orange aqua regia was agitated, a compound of chlorine was evolved from it which readily exploded the test.

15. Well stopped bottles containing Thomson's chloro-chromic acid, and chloride of sulphur, which had been made a considerable time, were opened, and the test being applied, it exploded repeatedly before it reached the fluids. In both fluids there seemed to be a partial decomposition. The chloro-chromic acid had a very strong odour of chlorine, and the gas in the bottle continued for some time to explode the test. On opening the bottle of chloride of sulphur, a quantity of the vapour and some of the fluid were forcibly expelled.

16. Being desirous of ascertaining to what extent common air might be mixed with chlorine gas, without impairing the action of the test, I made a number of experiments, using different proportions of chlorine and common air; in all of which the test readily exploded. I then collected in a cubic-inch measure divided into 100



parts,  $\frac{4}{100}$  of chlorine gas, which, by absorption was reduced to about  $\frac{1}{100}$ , the remaining  $\frac{22}{100}$  of the measure was now filled with common air, and being mixed, a bit of the test was dropped into the tube, when it readily exploded.

The foregoing experiments seem to prove that the new fulminating silver is a very delicate test of the presence of chlorine gas, nor does its delicacy appear to be impaired by exposure to the air, the light, or the sun's rays. Thus, some of the compound made last spring, was exposed on the sand bath during the summer months; part of it became of a dark brown, and part of a black colour. Some of the compound was exposed to the direct agencies of the sun's rays for some time, but the changes of colour it thus underwent, did not prevent it from instantly exploding in all cases in which it was put into chlorine gas, or into mixtures of this gas with other gases. I may also remark, that on exposing some of the test to the action of boiling water for some time, and then drying it, it exploded under the same circumstances as before.

The properties which appear to be regarded as most characteristic of chlorine, are its colour and odour. Though chlorine is easily recognised by its yellowish green colour; in cases when it is pure or nearly so, or when it forms the greater part of a gaseous mixture on which it does not act; yet it may, as is well known, be present in considerable quantity without exhibiting the least vestige of colour. Thus, in the common modes of making the gas, a considerable quantity must be generated, before any colour is apparent. And the purest chlorine, when mixed with a certain portion of common air, or other gases, on which it exerts no immediate action, is no longer distinguishable by its colour. Whereas the new test readily detects chlorine in the first bottle of air that comes over in the usual modes of making the gas; in cases when the gas is mixed with 0.98 or 0.99 of common air, and also, when even a solution of the gas in water is transferred from one bottle to another.

The *odour* of chlorine, though perhaps sufficiently characteristic, when the gas is mixed with other gases on which it exerts no action, or which have no powerful odour; yet it ceases to be so, when certain pungent gases or vapours are diffused through it. Thus, when a portion of chlorine was mingled with muriatic acid, or nitrous acid gas, or with the strongest liquid muriatic or nitric acid, the chlorine could not be satisfactorily distinguished; but in every instance of the kind the test exploded with flame an indefinite number of times. From a number of experiments I have made, I am disposed to regard what is commonly called the *odour* of chlorine, as a vague, and by no means a discriminative character, and that this odour exists in cases where we have no evidence of the presence of chlorine, or where, according to received opinions, it cannot exist. Thus, after exposing solutions of chlorine in water for several weeks or even months to the action of the sun's rays in summer, they are found still to have a strong odour which has been, I think, erroneously referred to chlorine.

I have hitherto said little concerning the specific action of chlorine gas, on the new

fulminating silver. The experiments I have made throw some light on the subject, while they afford additional proofs of the extreme delicacy of the test.

I filled a long narrow-necked matrass (smaller than a florence flask), with chlorine gas; placed it upright on a table, and successively dropped into it small portions of the new fulminating silver, until the number of separate explosions exceeded six hundred; when some fragments of the test on the table exploded, and the odour of chlorine was perceived. On examining the matrass, it was found sufficiently cracked to admit the gas to escape. In the course of this experiment, the explosions were uniformly accompanied with flame, and the appearance of a small dense white cloud. These phenomena, at first, occurred in the neck of the matrass, and part of the cloud sunk into the matrass, whilst the remainder rose into the air; but they took place, lower and still lower in the glass, as the number of explosions increased. The interior of the matrass was found covered with a finely divided dark purple substance, which readily dissolved in ammonia; and the solution treated with pure nitric acid in slight excess, gave a white precipitate, which melted at a dull red heat, and was chloride of silver.

In another experiment, a dry half ounce phial, having a narrow mouth, was filled with chlorine gas; small portions of the test were introduced, until one hundred and ninety-nine explosions had taken place. A little pungent vapour arose from the phial, and a peculiar odour was emitted, resembling that of chlorine in a state of great dilution. The vapour in the phial still possessed the property of bleaching; for it soon rendered moist litmus paper, white. A dark dove coloured substance remained principally at the bottom of the phial. It was chloride of silver, and there was a very minute portion of a crystallized substance attached to the upper part of the phial, which exhibited the properties of sal-ammoniac.

In a third experiment, a long dry tube of about the capacity of two cubic inches, was filled with chlorine gas, and the test was added until it ceased to explode. There was now distinctly perceived an odour precisely similar to that of the compound which is formed when fulminate of zinc is agitated in contact with chlorine gas. This compound is a yellow, oily, volatile fluid, resembling azotane in appearance, but having none of its explosive properties. Its odour is so acrid and peculiar that it can scarcely be mistaken. Its taste is sweetish, and astringent, with a certain degree of pungency, which remains for some time on the palate. It is apparently insoluble in water, but readily forms a sort of saponaceous compound with ammonia. It does not immediately redden litmus paper, but acquires this property after a short time. It is, I presume, a compound of fulminic acid and chlorine. There appears to be another compound of the same substances, but in different proportions. I obtained it by distillation after exposing the fulminating silver, and other analogous compounds, either diffused in water or dissolved in it to the action of chlorine gas. It is a colourless, transparent, and volatile fluid, having a peculiar and disagreeable smell and a taste at first

sweet, but which presently becomes sharp and enduring, somewhat resembling that of cayenne pepper. It is soluble in water, has no bleaching, but some acid properties, and may deserve farther examination.

It would seem from the foregoing results, that when the new fulminating silver is exposed to the action of chlorine gas, it is decomposed, chloride of silver is formed ; one part of the fulminic acid combines with that gas to form the peculiar compound just referred to, whilst the other part is decomposed, and affords by the reunion of its elements, sal-ammoniac. It seems probable, too, that carbonic acid gas and nitrogene, are at the same time evolved.

The action of chlorine gas on the new fulminating silver, is uniformly accompanied with flame ; this circumstance and the formation of ammonia above noticed, seem to favour the opinion I have advanced in the papers already referred to, that hydrogen enters into the composition of the fulminic acid.

The new fulminating silver, besides its use as a test for chlorine, might I think be employed with advantage as a substitute for Howard's fulminating mercury in the caps for percussion locks, which are now so much approved, and getting into such general use as threaten to supersede the common lock. The strong springs required in the percussion locks in which Howard's fulminating compound is used, are objectionable. The new fulminating silver requires much less percussive force to explode it than Howard's fulminating mercury ; nor is the effect of the explosion of the former, accompanied with that loud, and almost deafening report, of the latter compound.

From the known analogies existing between chlorine and bromine, the vapour of the latter might be expected to explode the test, as well as the former ; and this I find is the case. Thus, on putting a few drops of bromine into a small stoppered bottle, and dropping in a bit of the test ; it immediately exploded in the vapour, and the experiment was repeatedly tried at different intervals in the same bottle, with the same result. The test does not explode when brought in contact with iodine, either at the common temperature of the air, or when it is raised in vapour by heat.